1953 Report on Recommended Specifications For Microchemical Apparatus

Weighing and Drying

Committee on Microchemical Apparatus, Division of Analytical Chemistry, AMERICAN CHEMICAL SOCIETY

AL STEYERMARK, *Chairman, Hoffmann-La Roche Inc., Nutley, N. J.*
H. K. ALBER, *Arthur H. Thomas Co., Philadelphia, Pa.*
V. A. ALUISE, *Experiment Station, Hercules Powder Co., Wilmington, Del.*
E. W. D. HUFFMAN, *Huffman Microanalytical Laboratories, Wheatridge, Colo.*
E. L. JOLLEY, *Corning Glass Works, Corning, N. Y.*
J. A. KUCK, *College of the City of New York, New York, N. Y., and American Cyanamid Co., Stamford, Conn.*
J. J. MORAN, *Kimble Glass Co., Vineland, N. J.*
C. L. OGG, *Eastern Regional Research Laboratory, Bureau of Agricultural and Industrial Chemistry, Philadelphia, Pa.*
C. O. WILLITS, *Eastern Regional Research Laboratory, Bureau of Agricultural and Industrial Chemistry, Philadelphia, Pa.*

THE 1953 report of the Committee on Microchemical Apparatus concerns equipment for weighing and drying. The report includes recommendations for boats, a cup, weighing bottles, spatulas, forceps, tare bottles, and a modified Abderhalden drying apparatus. Recommended specifications for other pieces of microchemical apparatus have been published (*5*, *11*, *13–16*).

Questionnaires were sent to a representative number of chemists to obtain information on the equipment being used in the microchemical field. The replies were analyzed; items for which specifications appeared to be desirable were selected for this study.

It has been the policy of the committee to restrict consideration to items that are in fairly general use and have been described in the literature. As the work on weighing and drying equipment progressed, however, it became evident that several new items should be recommended and that several well-known pieces should be redesigned to such an extent that they can be considered new. These new and revised items were tested extensively by members of the committee and by collaborators before the final specifications were established.

Replies to the questionnaires also showed wide use of some equipment that does not appear to require attention by this committee. These items are: finger cots, capillary tubes, glass beads for tare flasks, mortars and pestles, capsules, small vacuum ovens, and various types of cloths made of chamois, cheesecloth, flannel, and silk.

Required dimensions for items for which specifications were thought to be desirable are given in the accompanying figures. Containers essential to the weighing operation, such as boats, are described in Figures 1, 2, and 3; a weighing cup in Figure 4, weighing bottle in Figure 5, and weighing bottle with outside cap in Figure 6. Several styles of spatula useful for introducing samples into the weighing containers are shown in Figures 7, 8, 9, and 10. Forceps suitable for grasping and manipulating the weighing containers themselves are shown in Figures 11 and 12. In the

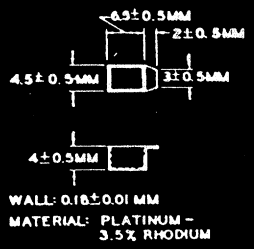


Figure 1. Combustion Boat, Size A

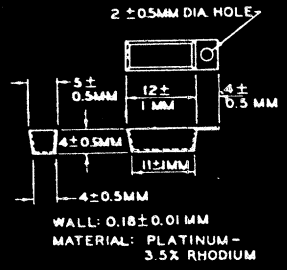


Figure 2. Combustion Boat, Size B

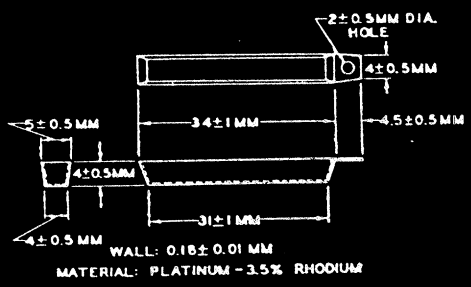


Figure 3. Combustion Boat, Size C

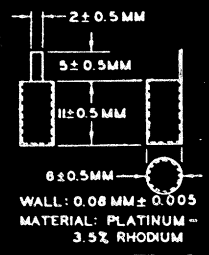


Figure 4. Weighing Cup

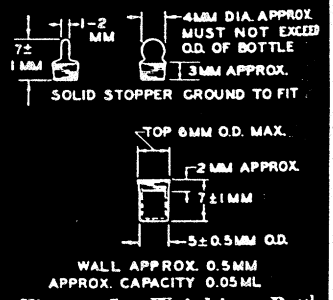


Figure 5. Weighing Bottle

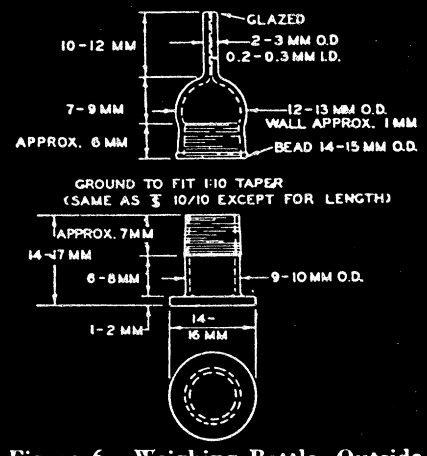


Figure 6. Weighing Bottle, Outside Cap

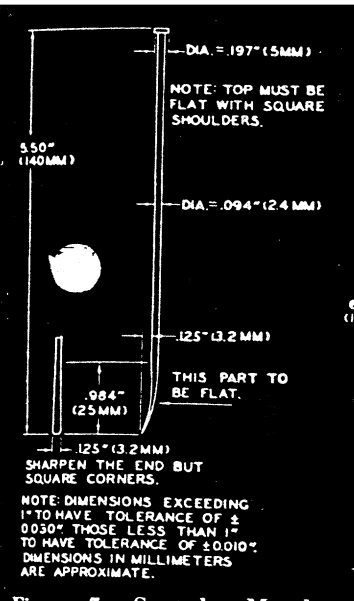


Figure 7. Spatula, Metal, Type A

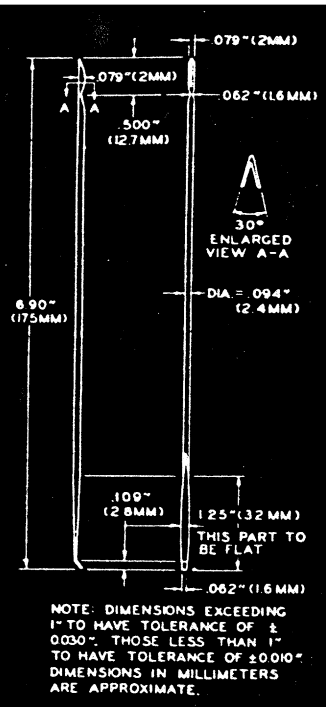


Figure 8. Spatula, Metal, Type B

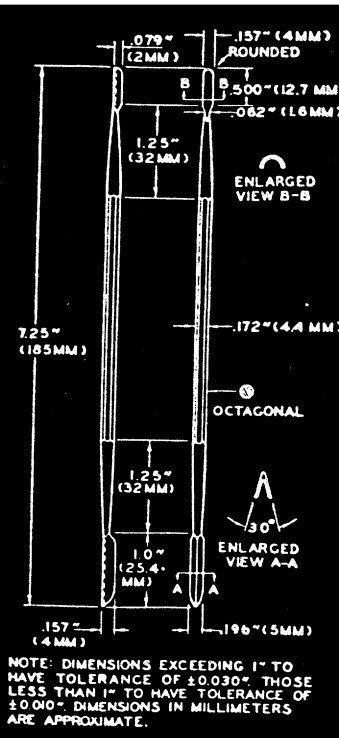


Figure 9. Spatula, Metal, Type C

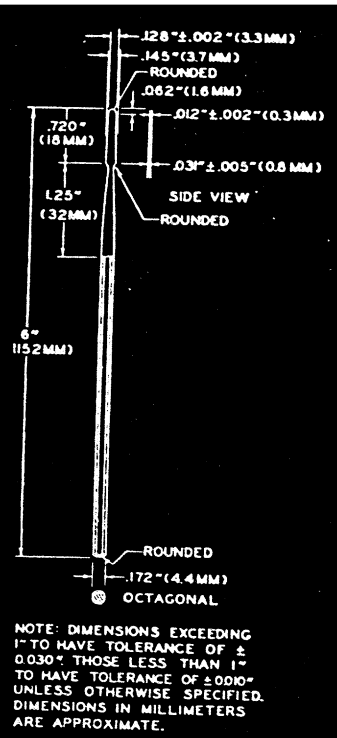


Figure 10. Spatula, Metal, Type D

drying operation, which usually involves weighing as well, one of several possible weighing bottles may be necessary. Figures 13, 14, 15, 16, and 17 show examples of suggested weighing bottles designed to accommodate the boats mentioned above and made from either metal or glass. Recommended tare bottles for counterbalancing and a modified Abderhalden apparatus for drying micro samples are shown in Figures 18 and 19, respectively. Detailed information regarding each item is given in the following paragraphs.

Combustion Boat. SIZE A. This boat (Figure 1), referred to in the literature as the Hayman boat (*9*, *12*), weighs approximately 0.45 gram. Because of its small size, it is used for samples weighing from 1 to 5 mg. For drying procedures, it should be used with the weighing bottle, pig type, metal, size A (Figure 15).

SIZE B. This boat (Figure 2) weighs approximately 0.7 gram and supersedes the boat for which recommended specifications are given in the 1949 report of the committee (*12*, *15*). It is the most commonly used size for handling samples in the range of 5 to 25 mg.

SIZE C. This boat (Figure 3) weighs approximately 1.5 grams and is used for semimicro purposes with samples that weigh up to 50 mg. and for micro purposes with bulky material or explosive substances. Capillaries containing [illegible] samples are placed in this boat, and the combination is introduced into a combustion tube.

Weighing Cup. This item (Figure 4) is designed as a sample container to fit into the weighing bottle, outside cap (Figure 6). This combination is useful for weighing hygroscopic materials.

Weighing Bottle. This type of weighing bottle was originally recommended by Roth (*7*, *10*). It may be inserted directly into such an item as a Carius combustion tube. It is to be made of borosilicate glass, so that it will withstand elevated temperatures and may be used repeatedly (Figure 5).

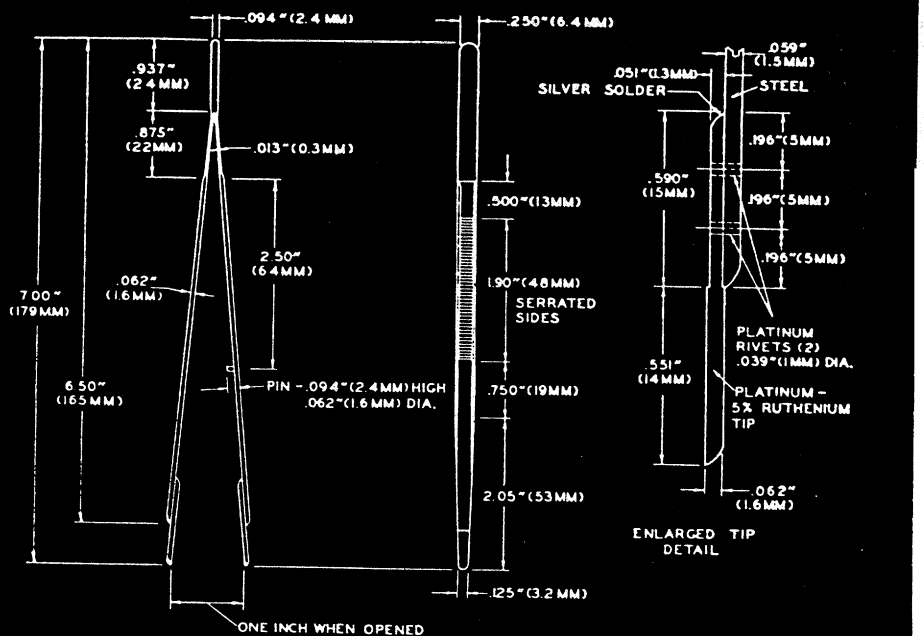


Figure 11. Steel Forceps (Nickel Plated) with Platinum-5% Ruthenium Tips

Weighing Bottle, Outside Cap. A series of experiments with hygroscopic materials, conducted by committee members, has established the following facts: that water vapor diffuses through a dry ground joint, that there is little difference in the amount of diffusion through a dry joint whether the cap (or stopper) is

closed or contains a capillary, either straight or with bulbs, and that in order to preserve a sample in the dry state, a lubricated closed cap is essential unless the vessel is to be stored in a desiccator. The use of lubricated ground joints on weighing bottles in general has been recommended by Benedetti-Pichler (*4*) and Bromund (*5*), in whose opinions this is the only way to obtain perfect seals. The use of stoppers with capillaries is also recommended by Benedetti-Pichler.

The micro weighing bottle originally described by Hayman (*6, 12*) meets the requirements outlined above and is the basis of the design (Figure 6) recommended in this report. It is to be made from soda-lime glass in order to reduce accumulation of static charges (*12*). The weighing cup (Figure 4) should fit inside for use as a liner, if so desired. The bottle has been designed with an outside cap, which permits the use of a lubricant with less danger of contaminating the sample than if an inside stopper were used. For the sake of simplicity, the capillary is straight.

Spatula, Metal. TYPE A. This type of spatula (*2, 12*) (Figure 7), in addition to being useful as a general spatula, can be used as a preparative tool, the bottom end for crushing crystals and the bent blade for scraping containers. It is to be made preferably of stainless steel.

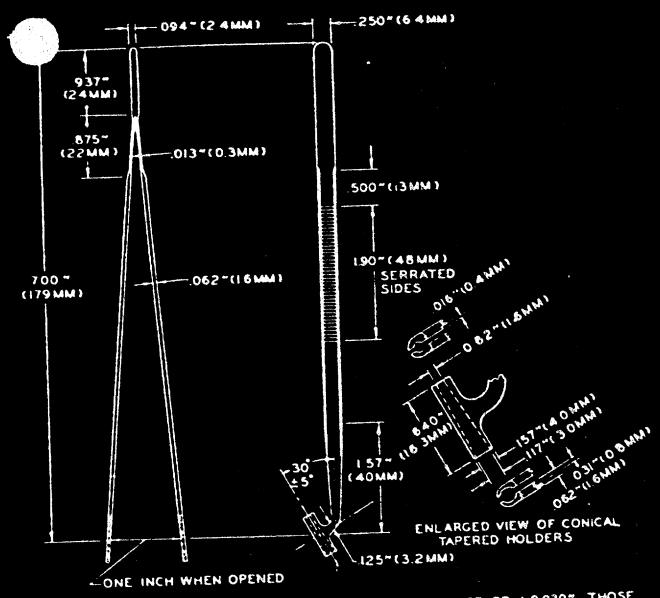
TYPE B. This spatula (*2, 12*) (Figure 8) has a flat, bent portion at one end, and a V-shaped scoop at the other. It is preferably made of stainless steel and is particularly useful in the weighing of samples.

TYPE C. The spatula (Figure 9) is suitable for the larger samples commonly encountered in semimicro and preparative work. It has a U-shaped scoop at one end and a V-shaped scoop at the other, and is preferably made of stainless steel. The spatula can be used for adding lead shot or beads to the tare flasks (Figure 18) and for measuring and introducing the solid reagent into the combustion tube of the apparatus for the manometric determination of carbon (*12, 17*).

TYPE D. This spatula (Figure 10), which is actually a dental spatula, has been found useful by members of the committee and others. It is made preferably of stainless steel.

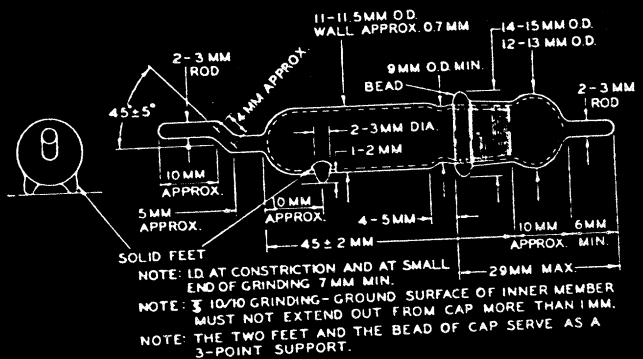
Steel Forceps (Nickel Plated) with Platinum–5% Ruthenium Tips. The available forceps with platinum tips (*12*) have not proved satisfactory because the platinum tips bend and eventually break off at one point of attachment. To correct this defect, the forceps have been redesigned.

The tips are now more sturdy and are made from the harder platinum–5% ruthenium alloy. A pin has been included. This serves as a stop to prevent the forceps



NOTE: DIMENSIONS EXCEEDING 1 INCH TO HAVE TOLERANCE OF ±0.030". THOSE LESS THAN 1 INCH TO HAVE TOLERANCE OF ±0.010". DIMENSIONS IN MILLIMETERS ARE APPROXIMATE.

Figure 12. Steel Forceps (Nickel Plated) with Conical Tapered Holders

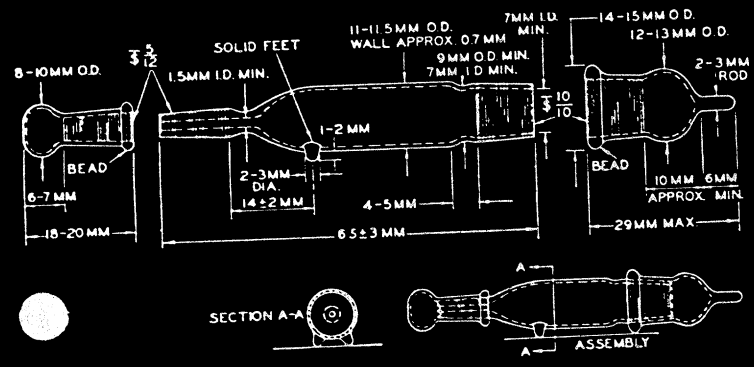


NOTE: I.D. AT CONSTRICTION AND AT SMALL END OF GRINDING 7 MM MIN.

NOTE: ⅗ 10/10 GRINDING-GROUND SURFACE OF INNER MEMBER MUST NOT EXTEND OUT FROM CAP MORE THAN 1 MM.

NOTE: THE TWO FEET AND THE BEAD OF CAP SERVE AS A 3-POINT SUPPORT.

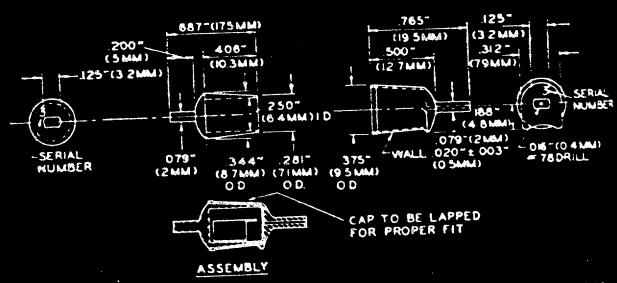
Figure 13. Weighing Bottle, Pig-Type, with Outside Cap



NOTE: THE TWO FEET AND THE BEAD OF THE LARGER CAP SERVE AS A 3-POINT SUPPORT

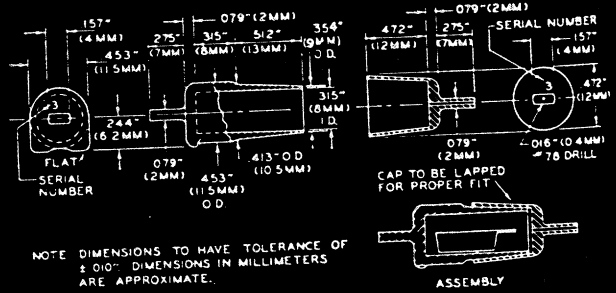
NOTE: ⅗ GRINDINGS-GROUND SURFACES OF INNER MEMBERS MUST NOT EXTEND OUT FROM CAPS MORE THAN 1 MM.

Figure 14. Weighing Bottle with Two Caps



NOTE: DIMENSIONS TO HAVE TOLERANCE OF ± .010" UNLESS OTHERWISE SPECIFIED. DIMENSIONS IN MILLIMETERS ARE APPROXIMATE.

Figure 15. Weighing Bottle, Pig-Type, Metal, Size A



NOTE: DIMENSIONS TO HAVE TOLERANCE OF ± .010". DIMENSIONS IN MILLIMETERS ARE APPROXIMATE.

Figure 16. Weighing Bottle, Pig-Type, Metal, Size B

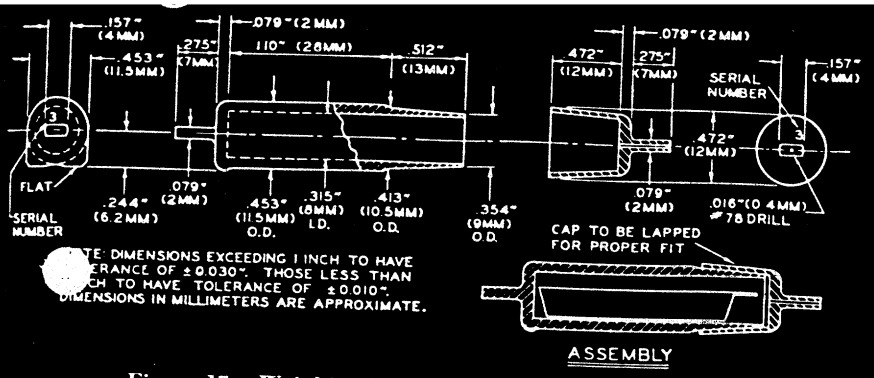
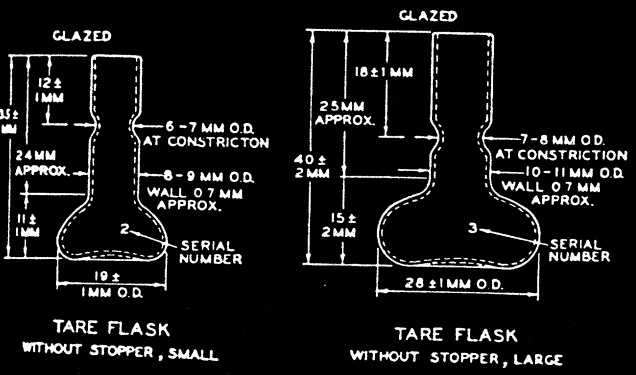


Figure 17. Weighing Bottle, Pig-Type, Metal, Size C



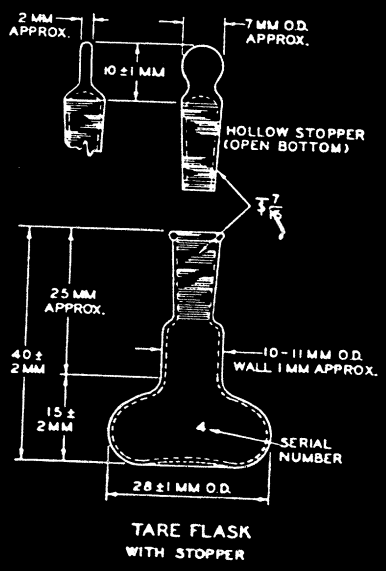


Figure 18. Tare Flasks

rom being depressed to such an extent that the ps can open, allowing the held object to drop. When the new forceps are pressed together, the ps make contact for a distance of 0.25 to 0.5 inch. Figure 11 shows the forceps with platinum–5% rhenium tips. The forceps may be made with- rare metal tips, using any other metal such as ckel-plated or stainless steel, depending upon eir intended use. The construction and over-all mensions should be identical to those shown in gure 11.

Steel Forceps (Nickel Plated) with Conical Holders. Until the introduction of the ceps (*7, 12*) of the type shown in Figure 12, ab- ption tubes, filter tubes, etc., were handled with e fingers covered with chamois cots, with metal s (*12*), or with forceps prepared by soldering ions of metal tubing to the tips of dissecting ceps. The forceps (Figure 12), which have been commercially available for a number of years, are of superior construction and provide a better means of handling objects. Because they are made in one piece from spring steel, they are also more durable.

Weighing Bottle, Pig-Type, with Outside Cap. This is the so-called Friedrich-type (*6, 12*) weighing bottle (Figure 13). It should be made preferably from soda-lime glass in order to reduce accumulation of static charges (*12*). The outside cap for the weighing bottle (Figure 6) may be used instead of the cap with the rod handle. The capacity of this bottle is approximately 3 ml.

Weighing Bottle with Two Caps. This bottle (Figure 14) should also be made preferably from soda-lime glass. The approximate capacity of the main body is 3 ml. This weighing bottle is used for extremely hygroscopic materials (*19*); only the small cap is removed for vacuum drying, and it is replaced as soon as the vacuum is released. For the determination of hydrogen or oxygen in extremely hygroscopic materials, the weighing bottle containing the combustion boat with sample is attached to a combustion tube by means of a rubber adapter, and the boat pushed into position in the combustion tube by passing a wire through the small joint. The outside cap for the weighing bottle (Figure 6) may be used instead of the cap with the rod handle.

Weighing Bottle, Pig-Type, Metal. SIZE A. This weighing bottle (Figure 15), referred to as the Hayman type (*9, 12*), is designed for use in conjunction with the boat, size A (Figure 1), as shown in the assembly drawing. The bottle should be made of an aluminum alloy, and the cap should be lapped to fit the body joint. The combination is useful for weighing and drying extremely hygroscopic material. Sample, boat, and bottle are weighed, and the combination, without removing the cap, is placed in the modified Abderhalden drying apparatus (Figure 19). During the drying operation, water leaving the sample passes through the capillary. The combination is finally removed from the dryer and again weighed. The advantages of a capillary have been noted under weighing bottle, outside cap

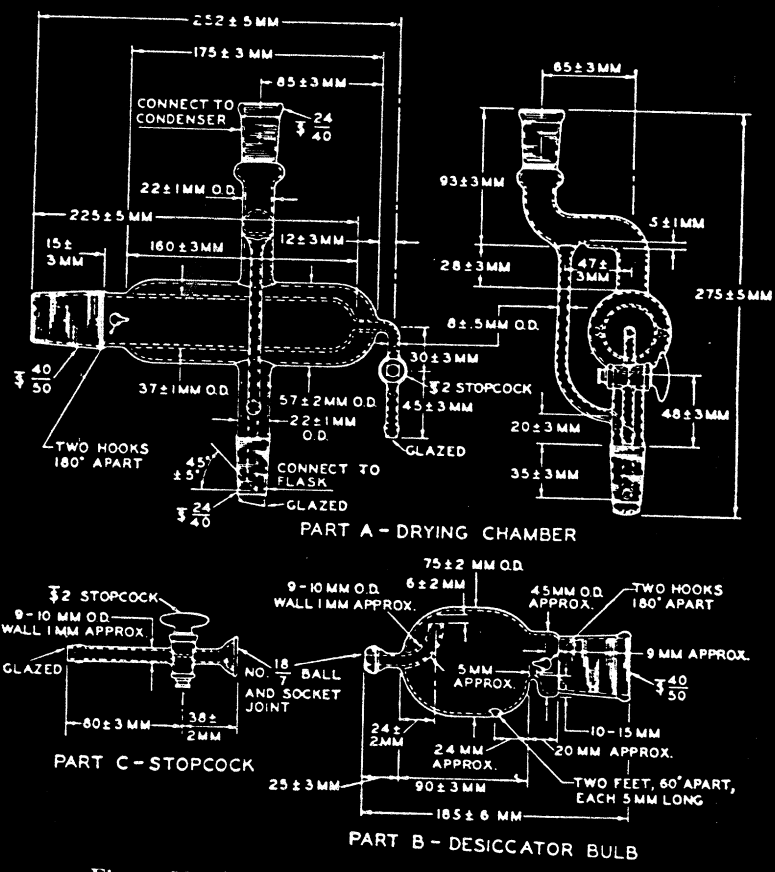


Figure 19. Modified Abderhalden Drying Apparatus

(Figure 6). The approximate capacity is 0.5 ml.; the approximate weight is 1.5 grams.

SIZE B. This weighing bottle (Figure 16) should be made from an aluminum alloy, and the cap should be lapped to fit the body joint. It is designed for use with the combustion boat, size B (Figure 2), as shown in the assembly drawing. The manner in which it is used is described under weighing bottle, pig-type, metal, size A. The approximate capacity of the bottle is 1 ml.; the approximate weight is 4.5 grams.

SIZE C. This weighing bottle (Figure 17) should be made of an aluminum alloy, and the cap should be lapped to fit the body joint. It is designed for use with the combustion boat, size C (Figure 3), as shown in the assembly drawing. The manner in which it is used is described under weighing bottle, pig-type, metal, size A. The approximate capacity is 2 ml.; the approximate weight is 6 grams.

Tare Flasks. Three types of tare flasks (*12*) are recommended, one with and two without a ground-in stopper (Figure 18). They should be made preferably of soda-lime glass (*12*). On all three flasks the serial numbers should be etched in order to avoid rough surfaces.

Modified Abderhalden Drying Apparatus. The committee, as well as others in the field, believed that the commonly used Abderhalden pistol dryers needed modification, particularly the desiccator bulb, which must be supported while removed from the main body. There was also danger of breaking the side arm when attaching rubber tubing to it, or on account of the weight of the tubing during actual use. The drying chamber has also been modified so that, in addition to the old practice of drying in vacuo, samples may be dried by the passage of dry air at reduced pressure, in accordance with the newer methods of drying (*18, 19*).

Figure 19 shows the modified Abderhalden drying apparatus intended to eliminate the disadvantages referred to above. The connection of the desiccator bulb to the vacuum by means of the ball and socket joint minimizes the risk of breakage and makes the desiccator bulb less unbalanced. The shape of the tube attached to the ball joint, in the desiccator bulb, is intended to prevent the desiccant from being carried over into the sample when the vacuum is broken. The tube at the left of the stopcock and to which the vacuum line is connected may be bent as desired. A cap for the ball joint and a stopper for the $\frac{40}{50}$ joint may be used to protect the desiccant when the desiccator bulb is temporarily disconnected and stands alone. The end view of part *A* (Figure 19) shows an upward indentation in the vapor tube located as near as possible to the reflux return. This prevents cooling of the drying chamber by cold condensate.

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Terminology for Describing the Performance of Analytical and Other Precise Balances

1954 Report and Recommendations of the Committee on Balances and Weights

L. B. MACURDY, *Chairman*, H. K. ALBER, A. A. BENEDETTI-PICHLER, HUGH CARMICHAEL, A. H. CORWIN, ROBERT M. FOWLER, E. W. D. HUFFMAN, P. L. KIRK, and T. W. LASHOF

THIS is a report and recommendation of the Committee on Balances and Weights in the Division of Analytical Chemistry of the AMERICAN CHEMICAL SOCIETY. This committee is a continuation of the earlier Committee on Microchemical Balances (*10*). The scope and name of the committee were broadened to include all precision balances and weights of interest to the chemist.

The new Committee on Balances and Weights held its first meeting on January 16, 1953, at the National Bureau of Standards, Washington, D. C. At this meeting it was decided that the committee would consider terminology for describing the performance of balances, procedures for testing balances, and the requirements of the chemist in the field of precision weighing. At its second meeting, January 22, 1954, also held at the National Bureau of Standards, the committee adopted the following report on terminology. This report, originally drafted by T. W. Lashof, was approved for publication in ANALYTICAL CHEMISTRY by the Executive Committee of the Division of Analytical Chemistry on March 29, 1954.

AT PRESENT there is no generally accepted terminology for describing the performance of analytical and other precise balances. This has caused much confusion in interpreting manufacturers' literature and purchasers' needs. The word "sensitivity" is the center of much of this confusion. The word has usually been used without being defined.

Sensitivity has been defined or used without definition in the following conflicting ways:

1. As the change in load in one of the pans required to change the swing—i.e., sum of turning points—by one division (*7, 8*).
2. As the change in load required to change the rest point by one division (*5, 6*).
3. As the change in load required to produce a perceptible change of indication: A perceptible change is sometimes defined to be $\frac{1}{4}$ division on swing for an analytical and assay balance